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**BIAXIALLY TEXTURED CONSTANTAN ALLOY
(Cu 55 wt%, Ni 44 wt%, Mn 1 wt%) SUBSTRATES FOR
YBa₂Cu₃O_{7-x} COATED CONDUCTORS (POSTPRINT)**

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Biaxially textured constantan alloy (Cu 55 wt%, Ni 44 wt%, Mn 1 wt%) substrates for $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ coated conductors

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Abstract

Commercially available constantan alloy rods (nominal composition Cu55–Ni44–Mn1 wt%) have been thermo-mechanically processed to develop biaxially textured substrates. It was found that the (001) recrystallization cube texture percentage could be increased from 72% to nearly 100% as the annealing temperature of the rolled substrates was increased from 750 to 1200 °C. A full width half maximum (FWHM) of 6.5° in (111) phi scans and an FWHM of 4.9° in (100) omega scans were observed in the substrates annealed at 1200 °C for 2 h. These substrates were found to have a Curie temperature of 35 K and so were paramagnetic at 77 K and ferromagnetic at 5 K with a saturation magnetization that is 2.5 times less than that of Ni–5 at.% W substrates. Yield strengths of highly textured constantan substrates were found to be 1.5 times that of textured pure Ni substrates at room temperature.

(Some figures in this article are in colour only in the electronic version)

1. Introduction

High temperature superconducting (HTS) coated conductors using $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ (YBCO) on buffered textured metallic substrates are being developed for a variety of power and energy applications [1–4]. A biaxially textured metallic substrate is essential in this technology to induce alignment in the YBCO grains so that conductors with a high critical current density can be made. The required alignment in YBCO grains is achieved through lattice matched buffer layers deposited on metallic substrates which also serve as barriers for metal and oxygen ion diffusion. This method of making coated conductor substrates is often referred to as the rolling assisted biaxial textured substrate (RABiTS™) approach [1].

When selecting an appropriate substrate material, the lattice match with buffer layers and the YBCO, ability

to develop biaxial texture, compatibility with subsequent multilayer depositions, yield strength, magnetic properties, and cost are among the important factors considered. Although pure Ni was initially used to demonstrate the feasibility of the RABiTS approach, the low yield strength and ferromagnetic nature of this material make it less suitable for processing long-length commercial coated conductors [5]. Alloying additions such as W or Cr to the Ni were made [6–9] to increase the strength and reduce the ferromagnetism without altering the lattice match considerably. An alloy of Ni–5 at.% W is the most common substrate material presently used in the textured substrate approach for making long-length coated conductors. In addition to developing good texture, Ni–5 at.% W has a reduced magnetic moment compared to Ni (about 50% less) and increased strength. While Ni–9 at.% W can reduce the ferromagnetic moment of the alloy significantly, obtaining the

texture in this alloy composition is very difficult [10]. Ni–Cr alloys with reduced ferromagnetic properties also have been demonstrated [11] to develop good biaxial texture, but problems with the formation of hexagonal Cr_2O_3 during the oxide buffer layer deposition inhibit the subsequent growth of high quality YBCO [7].

While copper or Cu–Fe substrates have better lattice match and have been shown [12] to develop good biaxial texture, they suffer from severe oxidation during the subsequent oxide-layer depositions. Even in very low vapour pressures of oxygen (10^{-6} Torr), the copper oxidation is stable [5]. To circumvent this, a buffer layer architecture involving nitrides [13] or metal coatings [14] has been proposed for use for copper substrates. However, the yield strength of annealed copper is too low to be useful to use as a practical substrate material for coated conductor technology. Hence there is a need for an alternative strong, non-magnetic copper alloy. By reducing the ferromagnetism of an alloy substrate, the magnetic hysteresis losses in coated conductors will be reduced. Although Ni–5 at.% W substrates are less ferromagnetic than Ni, they still have significant saturation magnetization (M_{sat}) of around 25 emu g^{-1} at 77 K. The cost of the coated conductor can be reduced if the substrate cost is less. This may be accomplished through the use of commercially available alloy compositions that are readily available.

In this study, commercially available alloys with good lattice match with YBCO have been investigated to explore the biaxial texture development. Two different alloys compositions of Cu–Ni–Mn were tested: manganin (Cu86–Mn12–Ni2 wt%) and constantan (Cu55–Ni44–Mn1 wt%). Although some initial observations of biaxial texture development in constantan alloy substrates were reported [15, 16], suitability for coated conductors was not evaluated. In this work, the experimental conditions such as annealing treatments were systematically varied to obtain improved texture. Magnetic properties, yield strength properties, etc were also measured and compared with similarly textured Ni or Ni–5 at.% W substrates and YBCO coatings with oxide buffers were deposited to evaluate the suitability of these substrates. Constantan is used in applications such as J-type thermocouples and strain gauges. Constantan alloys are used in making strain gauges due to high strain sensitivity, high resistivity, low temperature coefficient of resistance, good fatigue life, high elongation capability, etc. The nominal composition of constantan alloys is generally Cu55–Ni44–Mn1 wt% but this varies slightly with the manufacturer. The density is 8.9 g cm^{-3} with a melting point of 1225–1300 °C and the electrical resistivity is $\sim 52 \mu\Omega \text{ cm}$ at room temperature.

2. Experimental details

All the constantan and manganin rods used in the present study were acquired from GoodFellow. X-ray fluorescence analyses were done on the rods to determine the composition. The alloy rods were initially annealed at 450 °C in a reducing atmosphere of Ar/H_2 gas and then cold rolled at room temperature using hardened steel rolls. The rods were rolled with 10% reduction per pass to a total reduction of 99% to achieve a final thickness of the substrates of around $50 \mu\text{m}$. The substrates were then annealed in Ar/H_2 at various temperatures ranging from 750

to 1200 °C. For comparison of texture development in the manganin and constantan rods, both were heat treated at 750 °C for 1 h, and their texture was analysed by x-ray diffraction. Only the substrates which showed (200) textures (*c*-axis texture) in theta–two-theta scans were further given annealing treatments and then characterized using an orientation image microscope (OIM). OIM data were collected at several points on each sample with a scanning area of $450 \times 900 \mu\text{m}^2$. X-ray phi scans, omega scans and pole figures were also taken on a sample that was annealed at 1200 °C for 2 h that showed the best texture in OIM analyses. The XRD analysis was carried out using a Scintag XDS 2000 x-ray diffractometer equipped with a $\text{Cu K}\alpha$ x-ray source and a horizontal wide-angle four-axis goniometer. The full width half maxima (FWHMs) of the phi and omega scans were determined from a Gaussian curve fitted to the data.

The magnetic properties were determined by using a vibrating sample magnetometer (Quantum Design PPMS) at 77 and 5 K. Textured constantan substrates developed in-house were compared to textured Ni–5 at.% W substrates. Samples of approximately $10 \text{ mm} \times 10 \text{ mm}$ in size were carefully weighed and the magnetic response was measured as a function of applied field up to 2 T. In order to see how the saturation magnetization was changed by a Ni coating on the constantan, a $0.5 \mu\text{m}$ thick (nearly 1% by weight) Ni coating was sputter deposited on the constantan and also used for measurement. All the samples were mounted such that the applied field was parallel to the sample surface to minimize the demagnetization field effects.

The tensile strengths of textured constantan substrates were determined by using an in-house developed sample mounting method with an Instron machine. The details of the experimental method used are discussed elsewhere [17, 18]. The sample strength data were compared with those of annealed textured Ni substrates.

To evaluate whether oxide layers can be grown on these substrates, a buffer layer stack of CeO_2 , yttria stabilized zirconia (YSZ), CeO_2 with a subsequent YBCO layer was deposited using pulsed laser deposition (PLD). A Neocera chamber was used to deposit YBCO and other buffer layers using a Lambda Physic KrF ($\lambda = 248 \text{ nm}$) excimer laser. Typical processing parameters that were used for oxide buffers and YBCO depositions in PLD-coated conductors on Ni–5 at.% W substrates were used [4]. The superconductor samples were characterized by ac susceptibility and x-ray diffraction.

3. Results and discussion

X-ray fluorescence data revealed that the constantan alloy used in this study has the following composition: Cu $\sim 53 \text{ wt\%}$, Ni $\sim 45 \text{ wt\%}$ and Mn $\sim 2 \text{ wt\%}$, which is very close to the nominal composition of Cu55–Ni44–Mn1 wt%. Figure 1 shows theta–two-theta scans of the substrates rolled and then annealed at 750 °C for both the manganin and constantan substrates. It can be seen that peaks corresponding to other orientations are clearly present in the manganin but the constantan alloy substrates shows essentially *c*-axis ((200) reflection) texture. Since for a given set of rolling and annealing treatments

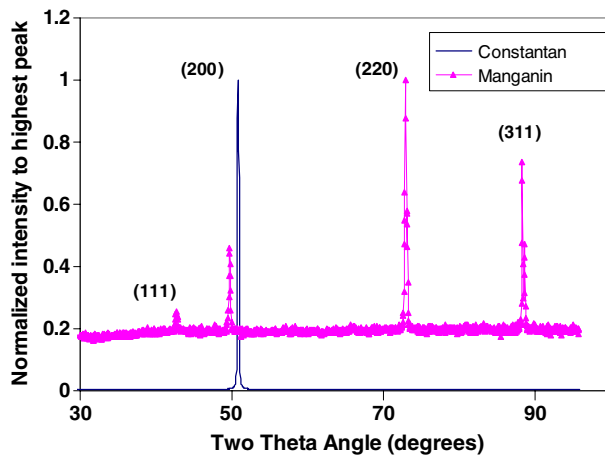


Figure 1. X-ray theta-two-theta scans of constantan and manganin rolled substrates annealed at 750 °C for 1 h.

constantan substrates showed indications of developing better texture, as compared to manganin, a detailed study of texture development in constantan alloy composition was conducted.

Figure 2 shows a series of orientation image microscope (OIM) images showing the grain boundary misorientations in

constantan alloy substrate samples treated for 1 h at different annealing temperatures, ranging from 750 to 1200 °C. It can be seen that the number of high angle grain boundaries ($>10^\circ$) (shown in black) decreases as the annealing temperature was increased. At 1200 °C most of the grain boundaries have misorientation of 5° or less. Figure 3 shows the colour-coded grain orientation for the same locations as shown in figure 1. It can be clearly seen that at the lower temperature the $\{111\}$ grains (blue) are present in larger amounts and the number density decreases as the annealing temperature is increased. At 1200 °C most of the grains are all with $\{100\}$ orientation except a few twins that were observed. Figure 4 shows the (100) pole figures obtained by using OIM data. It can be noticed that the texture greatly improves as the temperature is increased.

The variation of the $\{100\}$ $\{100\}$ recrystallization cube texture as determined by OIM was plotted as a function temperature and time (only at 1150 and 1200 °C) in figure 5. Increasing the annealing temperature from 750 to 1200 °C increased the percentage of cube texture (the fraction of $\{100\}$ plane orientation) from 72% to nearly 100% at 1200 °C. An increase in the annealing time from 1 to 2 h increased the percentage cube texture by 2–3% at 1150 or 1200 °C as shown in figure 5. However, the increase in time at these high temperatures also increases the grain size of the alloy which may reduce the yield strength. For this reason, the hold time at high temperatures was restricted to 2 h.

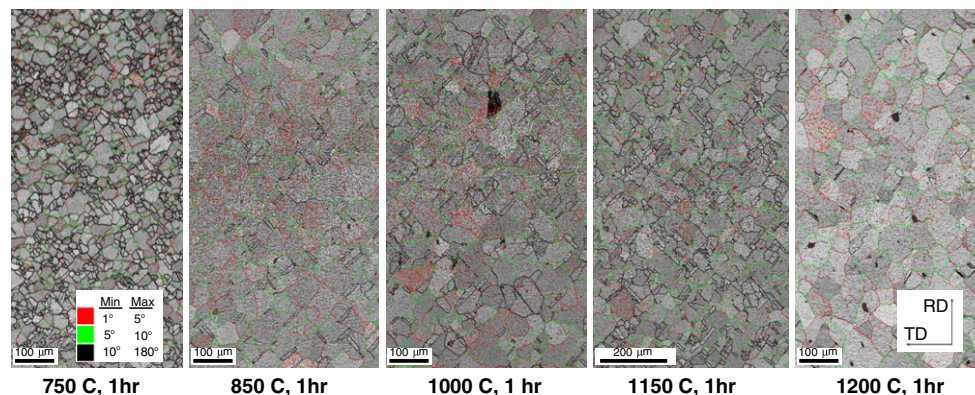


Figure 2. Orientation image microscope images taken on constantan samples treated at different temperatures showing the grain boundary misorientations.

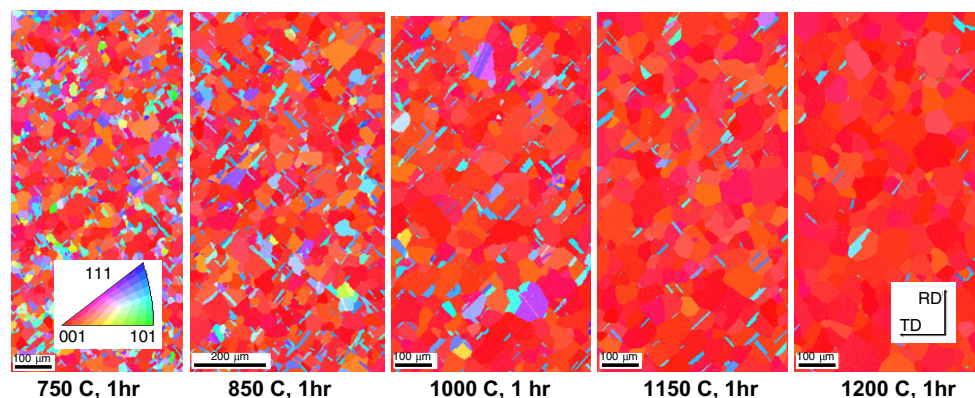


Figure 3. Colour-coded maps showing the inverse pole figures of the textured constantan substrates annealed at various temperatures.

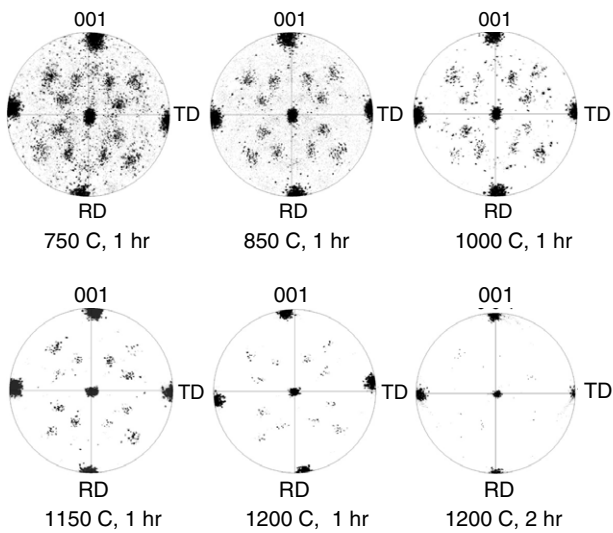


Figure 4. OIM (100) pole figures of the constantan substrates annealed at various temperatures.

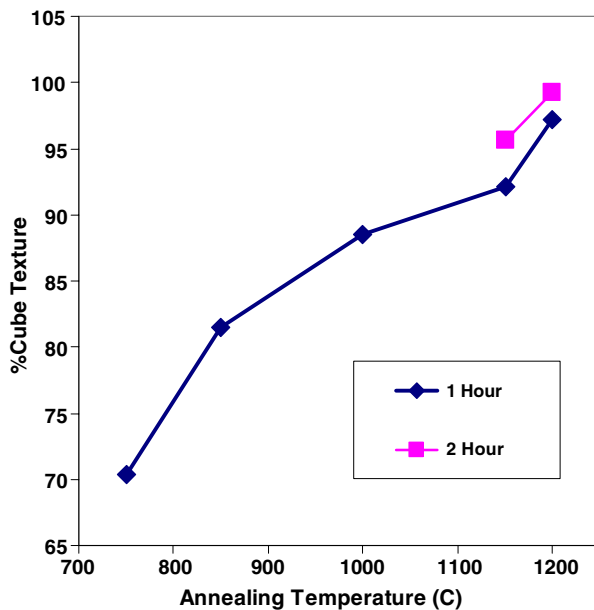


Figure 5. Increase in the percentage cube texture as a function of temperature and time.

Figure 6 shows the OIM data of a sample annealed at 1200 °C for 2 h which displays the grain boundary misorientation and grain orientation data taken from the same location. It can be seen that although the grain size is slightly increased to $\sim 100 \mu\text{m}$ as a result of the 2 h annealing time at high temperature, the texture is improved considerably. The constantan substrates annealed at 1200 °C for 2 h were further characterized by x-ray pole figures to confirm that the texture is present in the whole sample and not confined to small OIM observation areas. Figures 7 and 8 show typical x-ray {111} phi and omega scans taken on constantan samples annealed at 1200 °C for 2 h. A full width half maximum (FWHM) of 6.5° in the phi scan and FWHM of 4.9° in the omega scan was observed, indicating good cube texture in the sample. Figure 9

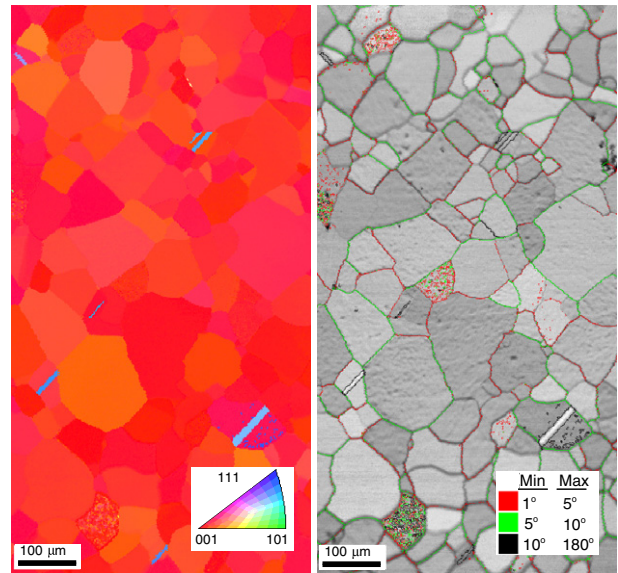


Figure 6. OIM data of a constantan sample annealed at 1200 °C for 2 h.

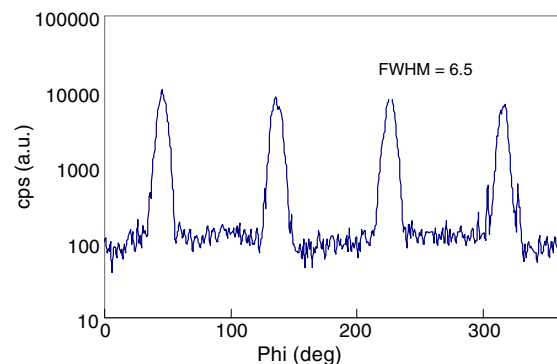


Figure 7. X-ray phi scans (111) of a constantan sample annealed at 1200 °C for 2 h.

shows the x-ray pole figures on a log scale, also indicating the presence of good texture and the absence of reflections from other orientations, corroborating the OIM data.

The Curie temperature was found to be around 35 K during the ac susceptibility measurements of a constantan substrate sample with a PLD-deposited YBCO coating. Figure 10 shows an ac susceptibility curve in an applied ac field of 0.025 Oe. The reduction in the shielding factor χ' below 35 K indicates that the localized strong field from the ferromagnetic grains in the substrate interferes with the intergranular coupling in the YBCO film. The grain boundary properties of the YBCO will need to be improved in order to diminish this effect and increase the film's critical current density as a direct consequence.

Figure 11 shows the summary of magnetization measurements taken on the substrates at 5 and 77 K. At 77 K, the constantan is paramagnetic but it is slightly ferromagnetic at 5 K. The paramagnetic magnetic moment of the samples at 77 K is 20% of the saturation moment of Ni-5 at.% W at 2 T and the saturation magnetization (M_{sat}) is 40% of the M_{sat} in Ni-5% W at 5 K. The absence of ferromagnetism at 77 K in constantan

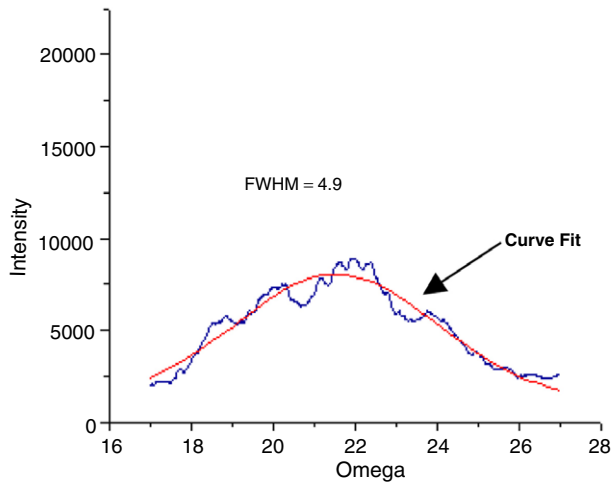


Figure 8. Omega scan (111) of the sample annealed at 1200 °C for 2 h.

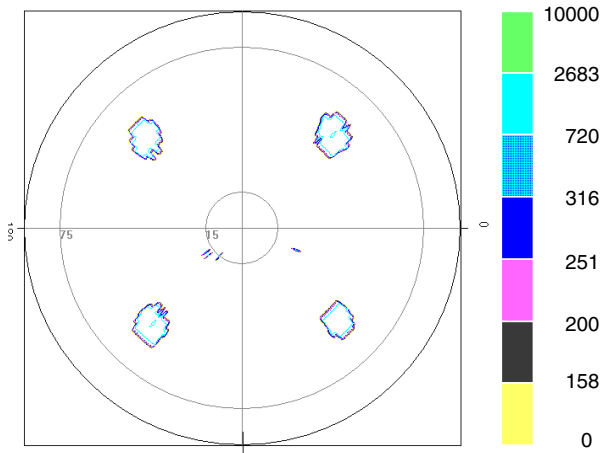


Figure 9. X-ray pole figures in log-log scale of a constantan sample annealed at 1200 °C for 2 h.

implies that the coated conductors made using these substrates could be free from magnetic hysteresis losses. Since the Curie temperature is around 35 K, the constantan substrates offer the flexibility of making the low ac loss conductors in the range of 35–77 K operation temperatures. The Ni layer on constantan results in an increase in magnetization roughly proportional to the Ni layer thickness, i.e. 1%.

The yield strength (YS) data of constantan substrates treated at 1200 °C for 2 h were found to be 74 MPa, as opposed to textured Ni which is around 52 MPa, using a 0.2% offset strain to determine the yield strength. More details of the test methodology and results can be found elsewhere [17, 18]. Although the YS is nearly 1.5 times than that of Ni substrate, the low value of 74 MPa is due to the high annealing temperatures used in this work. However, the substrates were found easy to handle during the subsequent depositions, unlike the textured Ni substrates that tend to bend easily during handling.

Initial results of YBCO coatings on buffered constantan substrates with a stack of CeO₂, YSZ, and CeO₂ buffer layers

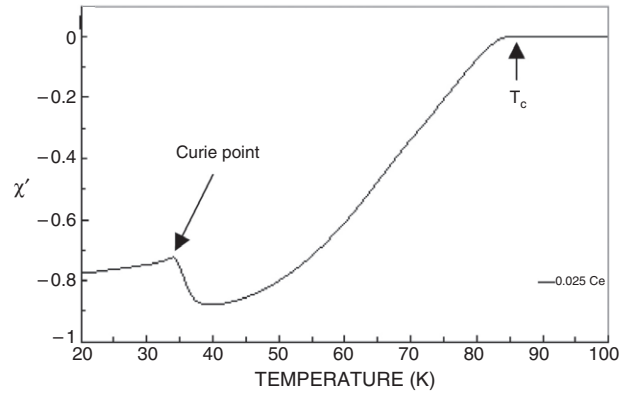


Figure 10. The ac susceptibility curve of a sample with YBCO coating deposited on a textured constantan substrate. The knee in the curve around 35 K is the Curie temperature of the substrate and the T_c onset temperature of YBCO around 85 K is also marked.

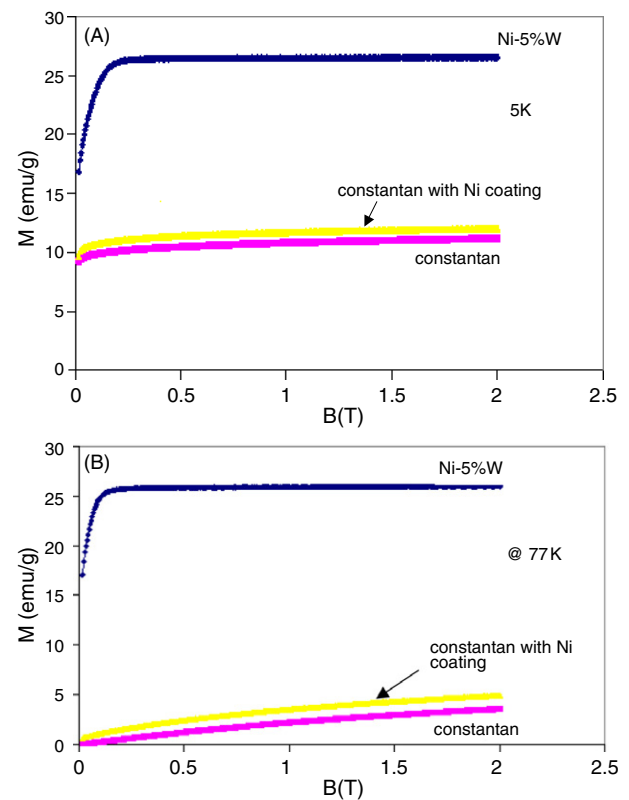


Figure 11. Saturation magnetization data taken at (A) 5 K, (B) 77 K of a constantan sample annealed at 1200 °C for 2 h compared with a well textured Ni and Ni-coated textured constantan substrate.

indicated that superconducting films can be grown on these substrates. As shown in figure 10, the T_c onset is around 85 K but the transition is wide, indicating that the critical current density (J_c) is low in these films. Although textured YBCO was grown (as determined by x-ray diffraction), the J_c was not high. To determine the cause of reduction in J_c of the films, the as-annealed substrates prior to deposition were tested for surface roughness by atomic force microscopy. The surface roughness (R_{MS}) was found to be high: 15–30 nm

in these samples. Typically a R_{MS} of 2–4 nm was obtained in Cu substrates when newly polished rolls were used [12]. It is thought that the high surface roughness of the substrates and the lack of other optimization processes that may be required for the YBCO deposition on these substrates are reasons for depressed J_c films. However, unlike the Cu or Cu–Fe substrates, the constantan substrates allowed adherent oxide buffer layers and YBCO depositions with PLD. Since a thin layer of Ni does not increase the ferromagnetic properties too much, a thin layer of Ni can probably be used to obtain higher quality YBCO. Further work to optimize the deposition conditions or utilization of nitrides or other metal coatings is underway.

4. Conclusions

Commercially available Cu55–Ni44–Mn1 wt% (constantan) alloy rods were thermo-mechanically processed to develop biaxially textured substrates. Nearly 100% cube texture was achieved when the rolled substrates were annealed at 1200 °C for 2 h. Substrates were paramagnetic at 77 K (hence no magnetic hysteresis losses) and less ferromagnetic than Ni–5% W at 5 K by a factor of 2.5. The yield strength of biaxially textured constantan substrates was found to be higher than that of biaxially textured Ni. A Ni sputtered coating did not increase significantly the saturation magnetization of the constantan substrate. Initial results of PLD-deposited YBCO and oxide buffers on these substrates, although with reduced quality, showed that the superconducting films can be deposited on these substrates with further efforts to optimize the depositions.

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